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Letter Report

Development of a Reliable Fabrication Technique to Produce Intermetallic Compounds

Contract No. N00014-85-C-0857

Prepared for:

Office of Naval Research
Arlington, VA 22219

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Quarterly Progress Report (#13)
(MCR-85-721)

1.0 Contract Number:
N00014-85-C-0857

2.0 Reporting Period:
8/10/89-11/9/89

3.0 ONR Scientific Officer:
Dr. Steven G. Fishman

4.0 Work Performed At:
Martin Marietta Astronautics Group, Denver, CO 80201

5.0 Principal Investigator:
Dr. William C. Moshier

6.0 Project Title:
Development of a Reliable Fabrication Technique to Produce Intermetallic Compounds

7.0 Description of Research:

7.1 Objectives of Present Research

- Intermetallic compounds offer very high specific material properties and property retention at elevated temperatures. However, reliable processing techniques to fabricate these materials have not been developed. This program will investigate the use of a novel fabrication technique involving a deformation-solid state reaction bonding process to form titanium beryllides. The objectives of this investigation are to:

  — Establish reaction kinetics for the formation of titanium beryllide intermetallic compounds.

  — Establish processing parameters needed to reproducibly fabricate these materials.

  — Characterize the properties of titanium beryllides fabricated using a deformation-solid state reaction bonding process.

  — Investigate the effects that alloying additions have on the crystal structure of titanium beryllides and determine how these changes impact the beryllide's mechanical properties.

7.2 Summary of Work Accomplished During Previous Reporting Period

- The Final Report (Dampening Characteristics of Metal Matrix Composites) was completed and delivered to ONR during the past reporting period.

- Fabrication of Metal Clad Filaments (MCFs)

  During the previous period, we had completed the first of two Ti-Be extrusions at Nuclear Metals, Inc (NMI). The extrusion consisted of Be wires that had been wrapped with Ti foil and packed into a steel can. The can was heated to 750°C (1400°C) and extruded through a 16:1 reduction die. The final diameter from this first extrusion, including the steel jacket, was 9.5 mm.

7.3 Progress During the Reporting Period

• Reaction Kinetics Study

A reaction kinetics study was initiated on the extruded MCFs. To prepare the samples, NMI etched off the steel can used in the extrusion, cut the extrusion into 0.75-in.-long samples, and repacked the samples into Ta-lined steel cans that were then evacuated. The samples were heat treated, either in a furnace or in a hot isostatic press (HIP), over a range of temperatures and pressures. Table 1 lists the conditions to which the samples were exposed.

Table 1. Aging Conditions for the Reaction Kinetics Study

Temperature (°C)	Time (hr)											
	0.5	1	2	4	6	8	12	18	20	24	100	200
850						x				x	x	x
975			x			x				x	x	
1000*					x		x	x		x		
1100	x	x		x					x			

* Hipped at 20,000 psi in Ar

The samples that were heat treated in the furnace were heavily oxidized. After sectioning and polishing the samples, the Be was found to have reacted extensively with the Ti. The reaction zone measured up to 100-μm-thick, and contains three individual layers (phases), with the layer closest to the Be being the thickest. The intermetallic region is heavily cracked, and did not appear to have been completely reacted at lower temperatures, i.e., it appeared as though there may be some un-reacted Be in the beryllide reaction zone. The Be, which still retained its extrude (round) shape, was completely separated from the intermetallic phase that had formed. In the case of the sample heat treated above the Ti-Be eutectic (~1050°C), the Be wires have settled to one side of the can, and an extensive reaction zone appeared around the Be. Unfortunately, the Ta also diffused into the Ti, and could not be removed from these samples.

The samples that had been HIPped also reveals an interface between the Ti and the Be. However, the Be was no longer round, but had been deformed during the reaction. The reaction zone between the Be and the Ti also contains three intermetallic phases, much like the material reacted in a furnace. However, the reaction zone does not contain cracks or other defects, there is no evidence of voids or disbonding at any interface, and the reaction zone appears uniform and completely reacted. The maximum thickness of the reaction zone for the HIPped samples is 125 μm.

The remaining material from this first extrusion was repacked in to a new steel can and re-extruded. A cross-section from this extrusion reveals that the Be fiber diameter now averages 300 μm. A total of four samples have been canned in evacuated Ta-lined steel cans and will be reacted in a HIP under the same conditions as the first extrusion. A section of the second extrusion was cut up into 2-in.-long lengths, de-canned, repacked into a steel can, and has been extruded a third time, so that the final Be fiber diameter is now 100 μm. This Be diameter will allow us to fully react the material in a HIP in a reasonable period of time. Both the samples from the second and third extrusions will be reacted in the HIP at the same time to reduce cost, i.e., one HIP run rather than two separate runs.

7.4 Tasks for the Next Period

- Reaction Kinetics Studies

Characterization of the reaction zone between the Ti and Be in the MCFs which began in this period and will continue into the next period. Measurement of the zone thickness for the HIPped samples has been completed using an Image analyzer, and the data is being evaluated to determine the growth kinetics of the reaction zone. The structure and composition of the reaction zone are being evaluated using X-ray diffraction, to characterize structure, and Auger electron spectroscopy, to analyze zone composition with a scanning Auger microprobe. This work has been initiated and is expected to be completed by the end of November.

The reaction of the second and third extrusion in the HIP will take place in early December. These samples will be used to study the condition in which the Be fiber is completely consumed during the reaction. In the study of the first extrusion, a significant amount of the Be remained after the reaction. These studies will focus on how the reaction continues once the Be is completely consumed, i.e., the reaction of the higher order, beryllium-rich beryllides to form lower order beryllides.

- Fabrication of Thin Films

Funding to perform the sputtering study reached Rocky Flats late in this quarter. A meeting to initiate the effort will occur on November 15 at Rocky Flats. It is expected that the first task, which involves sputtering pure Be onto Ti sheet, will be completed in early December. The revised schedule is listed as follows:

— Sputter deposit 50 ± 2 µm of Be on to a 406 µm thick Ti substrate over a surface area of 7.5 cm x 7.5 cm. A total of ten specimens are to be delivered to Martin Marietta Astronautics Group (MMAG) by 12/15/89.

— Fabricate a Ti-Be target so that a film with a composition of 30 ± 1 wt.% Ti and 70 ± 1 wt.% Be can be sputtered. Machining of the target will be complete by 12/21/89.

— Co-deposit a total of five Ti-Be films 50 ± 2 µm thick x 7.5 cm x 7.5 cm by 3/28/90.

Photographs of the target and a final report detailing the sputtering conditions used to fabricate the films will be delivered to MMAG no later than 4/30/90.

Once we have obtained these Be films, we will analyze their purity and microstructure. Reaction kinetics studies will be initiated on these samples. A second study that will involve determining the effect of roll bonding the Be-Ti foil will also be initiated to obtain processing information. Several of the foils will be sectioned, cleaned, and stacked inside a Ta-lined steel can. The can will then be evacuated, sealed, and hot rolled to provide a laminated structure. These samples will be reacted to form beryllides after hot rolling.

7.5 Presentations During This Period

- None

7.6 Technical Reports During This Period

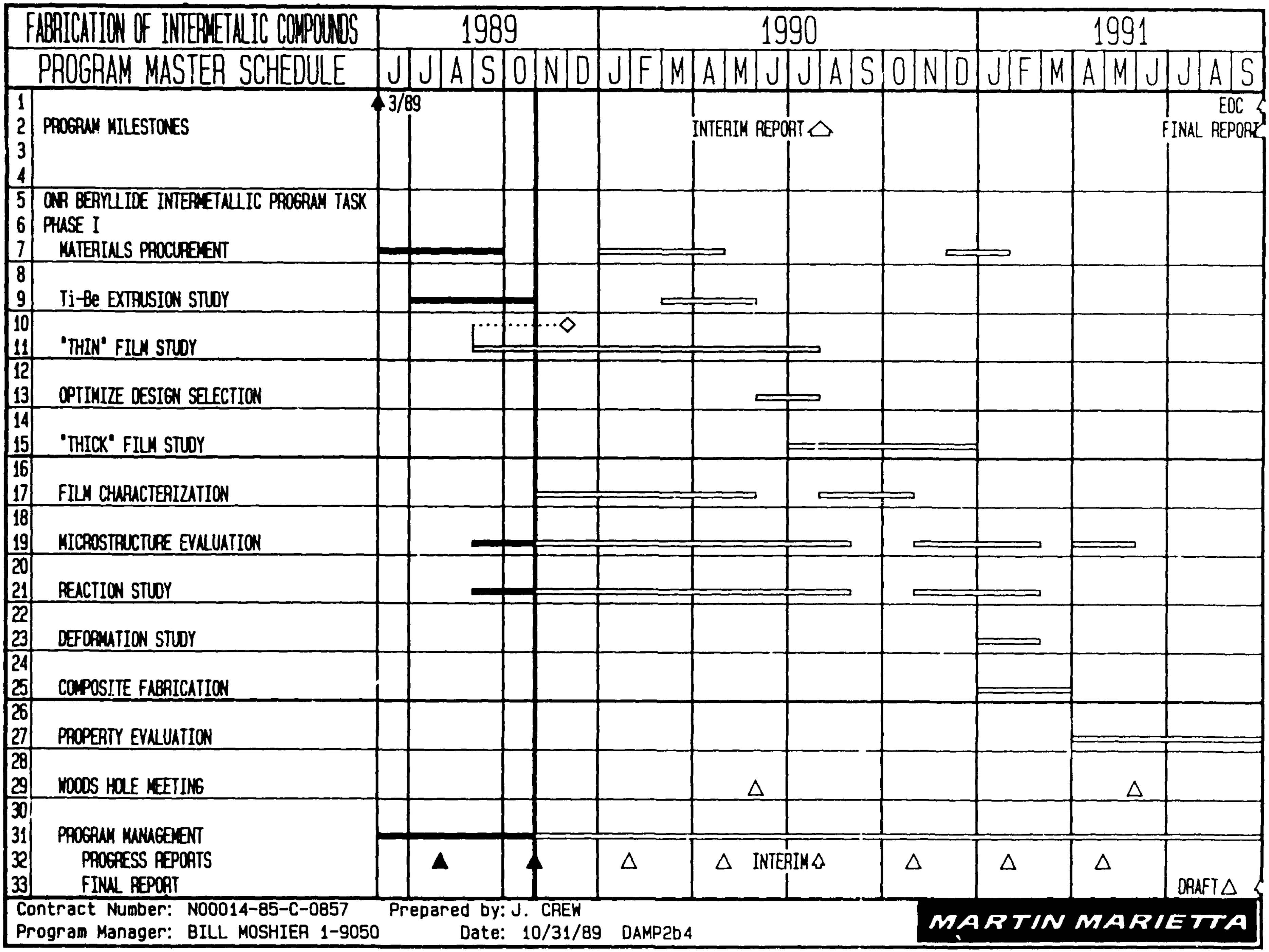
- "Damping Characteristics of Metal Matrix Composites," S.P. Rawal, J.H. Armstrong, and M.S. Misra, Report No. MCR-89-506, May, 1989

7.7 Publications During This Period

- None

7.8 Participants On The Program (To Date)

Name	Task
Nuclear Metals Inc., Concord, MA	Fabricate MCf/Extrusion/Specimen Prep
EG&G, Rocky Flats, Golden, CO	Sputter-deposit Be/ Ti-Be films
Colorado School of Mines, Golden, CO	TEM/Hot Rolling
Los Alamos National Laboratory, Los Alamos, NM	TEM/Metal Working
Coors Ceramic Co, Golden, CO	HIP Facility
Martin Marietta Laboratories	HIP Facility


FABRICATION OF INTERMETALIC COMPOUNDS
PROGRAM MASTER SCHEDULE
1989
1990
1991
J J A S O N D J F M A M J J A S O N D J F M A M J J A S
1
2 PROGRAM MILESTONES
3
4
5 ONR BERYLLIDE INTERMETALLIC PROGRAM TASK
6 PHASE I
7 MATERIALS PROCUREMENT
8
9 Ti-Be EXTRUSION STUDY
10
11 'THIN' FILM STUDY
12
13 OPTIMIZE DESIGN SELECTION
14
15 'THICK' FILM STUDY
16
17 FILM CHARACTERIZATION
18
19 MICROSTRUCTURE EVALUATION
20
21 REACTION STUDY
22
23 DEFORMATION STUDY
24
25 COMPOSITE FABRICATION
26
27 PROPERTY EVALUATION
28
29 WOODS HOLE MEETING
30
31 PROGRAM MANAGEMENT
32 PROGRESS REPORTS
33 FINAL REPORT
3/89
INTERIM REPORT
EOC
FINAL REPORT
INTERIM
DRAFT
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